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X A LABORATORY METHOD FOR FILLING AEROSOL CONTAINERS

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The filling of containers with liquefied-gas aerosols for experimental purposes has always presented certain difficulties, chief among which are inaccuracies in weighing and contamination by moisture. The contamination of hexaethyl tetraphosphate aerosols by moisture may lead to serious corrosion problems and the formation of solid particles which will clog the nozzles. An accurate laboratory method has been developed which overcomes these difficulties and is suitable for filling all sizes of containers and for all types of standard formulas.

Most of the liquefied-gas aerosols in use contain Freon-12 (dichlorodifluoromethane) as the propellant. This liquid boils at  $-21.7^{\circ}$  F. ( $-29.8^{\circ}$  C.) and has a gage pressure of 67.4 pounds per square inch at  $68^{\circ}$  F. ( $20^{\circ}$  C.). Methyl chloride, which is used in the hexaethyl tetraphosphate aerosols, boils at  $-10.7^{\circ}$  F. ( $-23.7^{\circ}$  C.) and has a gage pressure of 56.32 pounds per square inch at  $68^{\circ}$  F. ( $20^{\circ}$  C.). Methyl chloride is not suitable, safe, or recommended for use in general-purpose aerosols and should be used only where due precautions are taken to protect the operator.

Method

The predetermined amounts of pyrethrum extract, DDT, or other insecticides plus any solvents needed are first weighed, and the solution is warmed, if necessary, to dissolve all the material. The container to be filled is washed several times with acetone. Many of the refillable containers have a 1/8-inch brass pipe plug in the bottom for use in cleaning and filling. The 5-pound containers are washed by evacuation, drawing in 200 to 300 ml. of dry acetone, and shaking. The acetone is then removed by connecting the valve to a compressed-air supply and forcing the acetone from the container under pressure. This process is repeated until the acetone from the container is clear. The container is then dipped in hot water and connected to a vacuum pump for 30 minutes. A short length of copper tubing with a flared fitting is attached to the evacuated container. The weighed solution containing the insecticide and solvent is then drawn in. Care must be taken not to admit air, because this will increase the initial pressure of the system and after a portion of the aerosol has been dispensed the pressure will drop, thus decreasing the rate of flow of the aerosol through the nozzle.

When many bombs are being filled with the same formula, the equipment shown in figure 1 is used. The aerosol concentrate is placed in

the reservoir (A). The measuring flask (B) is calibrated for the solution being used. To prevent moisture from being absorbed by the solution, calcium chloride tubes (C) are placed in the stoppers of both the reservoir and the measuring flask.

To fill the aerosol container (not shown) the aerosol concentrate is first measured in flask B. The dried container is then evacuated and connected to the copper tube from the measuring flask by means of a refrigeration quick coupler (D). The contents are slowly drawn into the container. When the liquid is near the bottom of the flask, care must be taken to avoid air being drawn into the container. The container is then disconnected and placed on the balance for the filling operation.

The filling apparatus shown in figure 2 was designed to eliminate the possibility of injury from the escaping propellant liquefied gas at the end of the operation. Electric solenoid valves insure positive cutting off of the liquefied gas when the desired amount has been measured into the container.

After the concentrate has been added, the aerosol container (G) is placed on the balance (H) and connected to the flexible refrigeration hose (F) with the container valve closed. Solenoid valve C is then opened by turning the switch at E to fill the system, including the hose, with the propellant. The container is then weighed, and additional weights equivalent to the amount of propellant to be added are placed on the balance pan.

The propellant reservoir (A) is then heated with the steam jacket (B) and the valve on the aerosol container is opened to allow the propellant to flow in. The container is under partial vacuum, and until the natural boiling point of the propellant is reached there will be a pronounced cooling of the container. When the desired amount has been added, the switch for valve C is turned off to stop the flow of the propellant to the container. The valve on the aerosol container is then closed and solenoid valve C-1 is opened. A 5-pound aerosol container (I) is then placed in a container (D) of dry ice to cool it below the natural boiling point of the propellant, thus acting as a vacuum pump. By this means all the liquid in the filling system is removed and collected in the container I. Valve C-1 is left open for approximately 5 seconds and then closed. The aerosol container (G) may then be disconnected without danger of freezing the hand or having liquid spurt in the eyes. The complete operation for filling a 5-pound aerosol container requires approximately 30 seconds.

#### Equipment Needed

- 3 - 1/4-inch SAE 7/16-20 thread quick couplers.
- 2 - solenoid valves, 3/16-inch orifice Detroit Lubricator Co. 115-230 v., a.c., No. 683-3, or equivalent.
- 1 - double wall switch
- 1 - laboratory balance, 10 kilograms capacity
- 3 - 1/4-inch flexible refrigeration hose, 24 inches long.
- 10 ft. of copper tubing.

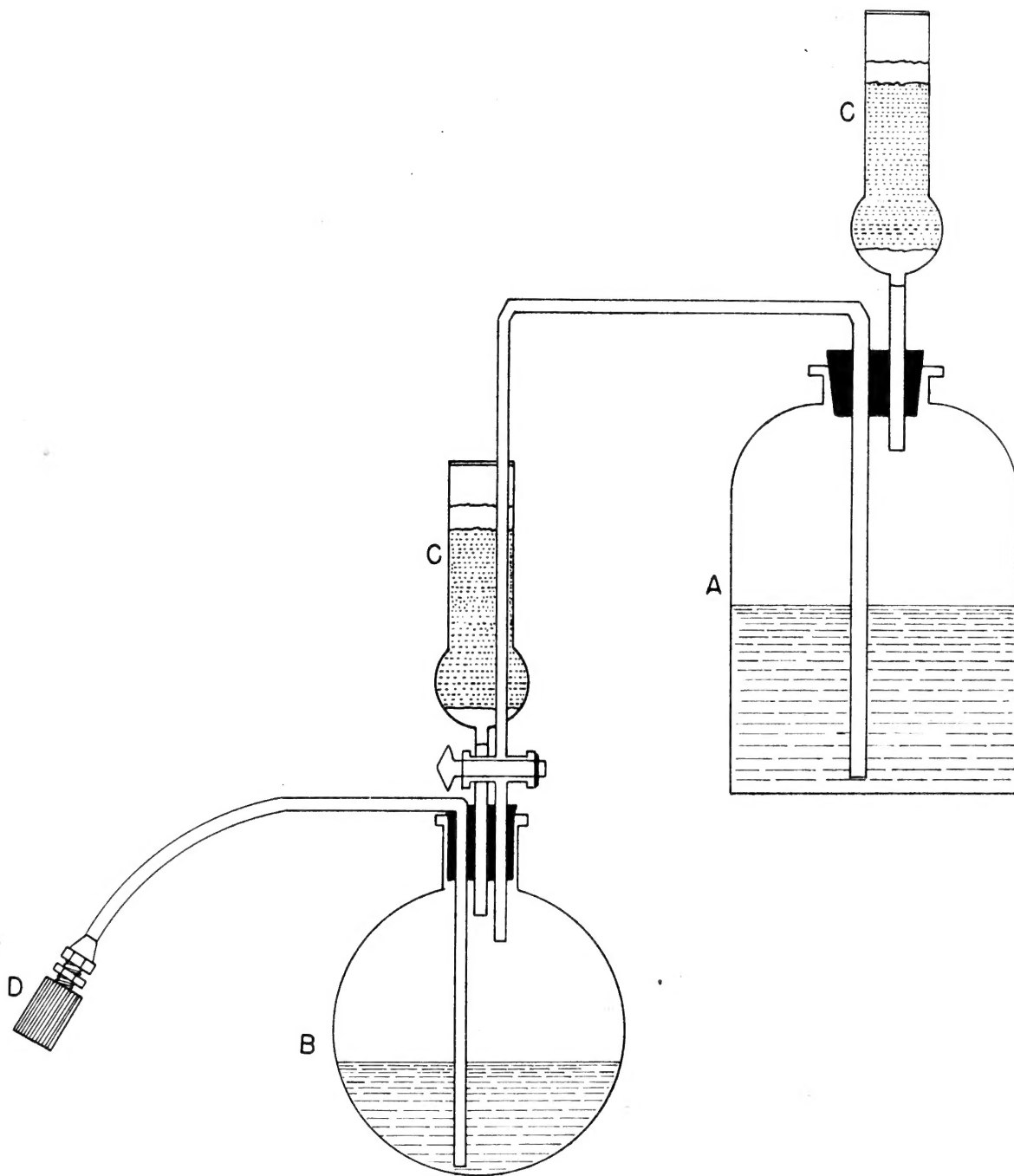


Figure 1.--Calibrated apparatus for aerosol concentrates.



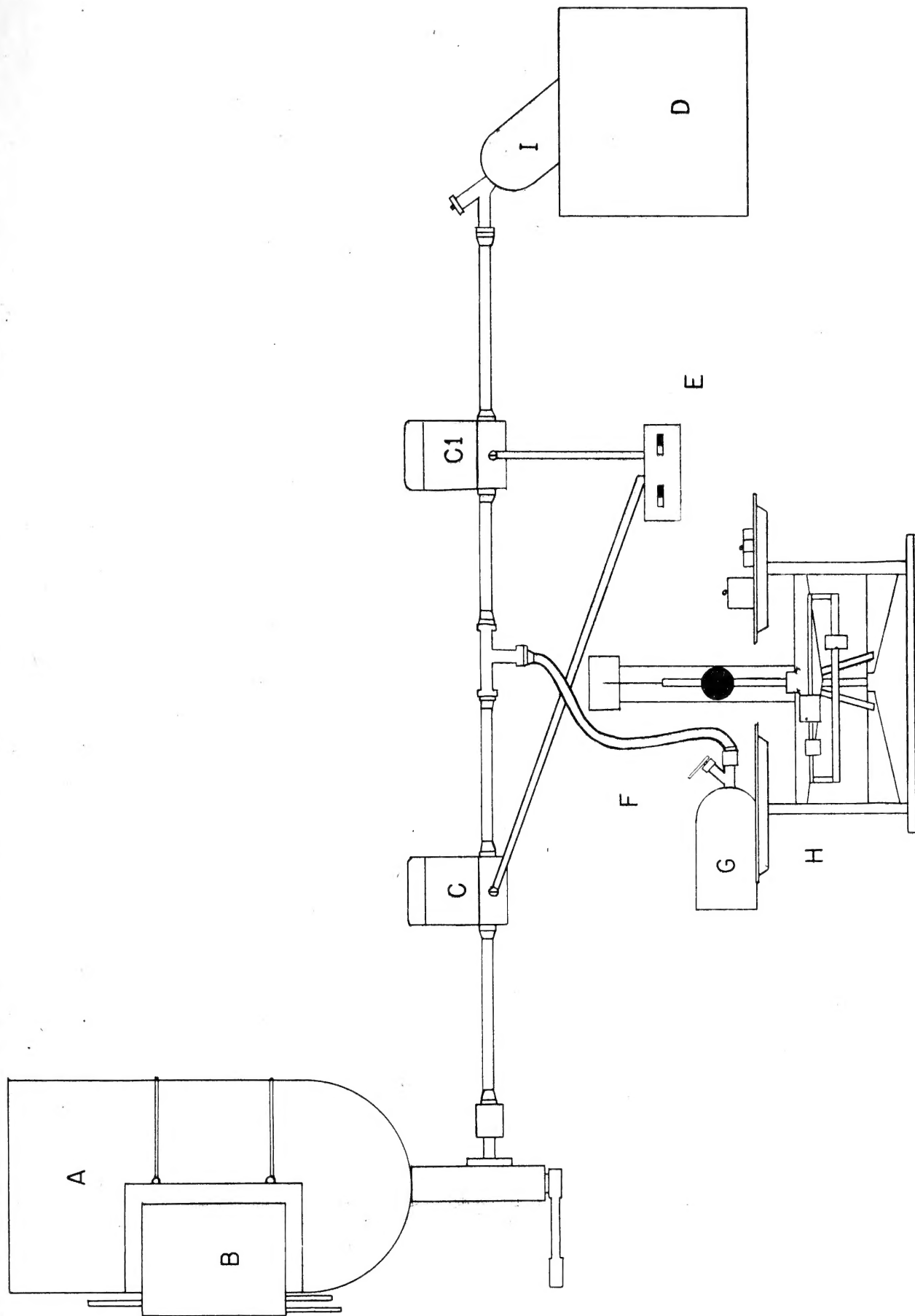


Figure 2.--Apparatus for filling aerosol containers with the propellant liquid.

